THERMAL CONDUCTIVITY MEASUREMENT OF SUB-MICRON THICK FILMS DEPOSITED ON SUBSTRATES BY MODIFIED AC-CALORIMETRY (LASER-HEATING ÅNGSTROM METHOD)

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ABSTRACT

Technological development, especially in microelectronics, have necessitate the development of new and improved methods of measuring the thermal properties of materials especially in the form of ultra-thin films.

Previously, modified ac-calorimetry (laser-heating Ångstrom method) using a scanning laser as the energy source has been developed and shown to provide accurate values of thermal diffusivity and derived thermal conductivity for broad range of materials in the form of free-standing thin sheet or film, wires including fiber bundles and some films on substrate.

This paper describes further applications of the modified ac-calorimetry for the measurement of thermal conductivity of thin films deposited on substrates. It has been used to measure successfully the thermal conductivities of 1000 ~ 3000 Å thick aluminum nitride films and aluminum oxide films etc., which were deposited on a glass substrate. The method was also applied successfully to the thermal conductivity measurement of sub-micron-thick chromatic films deposited on PET substrate, which are photo-thermal recording layers, used in the media of CD-R drives of the computer system.

KEYWORDS

ac-calorimetry, aluminum nitride, aluminum oxide, CD-R, thermal conductivity, thermal diffusivity, thin film, Ångstrom method•

1. INTRODUCTION

In many high power density electronic components many materials are used in the form of single and composite thin films, many of which may have thermal conductivities that differ from those of bulk materials due to their reduced thickness. These issues require that thermal properties be measured on the particular form and under conditions in which the material is applied. This requires the use of newer measurement methods than those that have been used conventionally for bulk materials.

Modified ac-calorimetry (laser-heating Ångstrom method) using a scanning laser as the energy source has been shown to provide accurate values of thermal diffusivity and derived thermal conductivity for broad range of materials in the form of free-standing thin sheet or film, wires including fiber bundles and some films on substrate. [1, 2] The method has been verified by using standard reference materials of $500 \sim 700 \,\mu m$ thick, which are also measurable by other traditional method such as laser flash method and/or guarded-hot-plate method. [3, 4]

This technique has now been applied to the measurements of thermal conductivity of sub-micron thick films deposited on a substrate• from the measurement of the apparent thermal diffusivity of the composite and of the thermal diffusivity of the substrate alone. The major advantages of this development are:

(1) The sensitivity of the measurements of thermal conductivity of deposited films becomes higher as both the thermal conductivity and the thickness of the substrate reduces. The method is applicable to any thermal conductivity and film thickness and measurements can be made without error due to heat loss from the surface of the specimen.

- (2) The precision of the thermal diffusivity of the free-standing specimen has to be much higher than that of required precision for the thin film. The method is capable of measuring free-standing specimen with a precision of ± 0.3 %.
- (3) As there are no standard reference materials of sub-micron films, the method must be applicable not only to thin films, but also thick films. The method is applicable for broad range of thicknesses from 1 to 700 μm .

It has been found that commercially available glass sheets do not have overall homogeneity throughout a whole sheet or lot and as a result the thermal conductivity can differ by 3 %. This uncertainty of the thermal conductivity can cause large error on the measurement of composite films. To solve this problem, a differential method has been developed in which the thin film is deposited on a half surface of the rectangular substrate to ensure the homogeneity of the substrate material between the deposited region and the non-deposited region. [5]

The method has two major applications of thermal conductivity measurement of thin films deposited on substrates. The first case is that the thermal conductivity measurement of electrical-insulating layers in electronic devices, such as AlN films or Al₂O₃ films used in MR or GMR sensor head of the hard disk drive of the computer system. In this case, normal-to-plane thermal conductivity is essentially important and its thermal conductivity is required to be as high as possible to obtain good heat dissipation. The second case is that thermal conductivity measurement of thin films, which are photo-thermal recording layers, used in the media of CD-R, CD-RW, DVD and MO drives of the computer system. In this case, in-plane thermal conductivity is essentially important and its thermal conductivity is required to be as low as possible to obtain higher density of memory. The present method is most suitable for the latter case,

because the method measures in-plane thermal diffusivity and/or thermal conductivity. Furthermore in the latter case plastics substrates are commonly used and these have a much lower thermal conductivity than the glass, providing a potentially higher sensitivity of thermal conductivity measurement of thin films deposited on the substrates.

2. THEORETICAL CONSIDERATIONS

The principle of the measurement method is shown schematically in **Fig. 1**. A portion of a rectangular film specimen is heated by a modulated laser beam, which is a line heat source, made by scanning the laser spot rapidly with constant speed in the width direction of the specimen. [6, 7] Temperature waves, which propagate one-dimensionally in the long direction of the specimen, are produced. As the irradiated portion of the specimen x changes, the ac temperature response is measured by a small thermocouple attached at the opposite face of the specimen. [8, 9] By analyzing the amplitude decay and the phase shift of ac temperature as a function of the spatial distance between the irradiated portion and the temperature sensor, the logarithmic decrement of the amplitude, k_a (reciprocal of the diffusion length) and phase shift increment, k_p (wavelength) can be obtained.

In the special case when no heat loss from the surfaces exists, k_a , k_p coincide each other and are equal to k. However in the general case when heat loss from the surfaces exists, k_a , k_p do not coincide each other and are not equal to k, but the square root of the product of the both values is equal to k. as shown in equation (2). By using k_a , k_p and the frequency f, according to the equation (1) and (2), it is possible to determine the true thermal diffusivity, D of the specimen. [10, 11] By selecting the appropriate frequency

the thermal diffusivity of wide variety of film materials from CVD diamond to polymers with a thickness of $1 \sim 700 \, \mu m$ can be obtained.

$$k = \sqrt{\frac{\pi f}{D}} \qquad (1) \qquad \qquad k = \sqrt{k_a \times k_p} \qquad (2)$$

A schematic of the setup for a thin film deposited on a half surface of the rectangular substrate is shown in **Fig. 2**. The apparent thermal diffusivities of the deposited region (thin film + substrate), D_{012} and the non-deposited region (substrate), D_{01} is given by equation (3) and (4) respectively.

$$D_{012} = \frac{D_0 C_0 d_0 + D_1 C_1 d_1 + D_2 C_2 d_2}{C_0 d_0 + C_1 d_1 + C_2 d_2}$$
(3)

$$D_{0I} = \frac{D_0 C_0 d_0 + D_1 C_1 d_1}{C_0 d_0 + C_1 d_1} \tag{4}$$

Where, suffix 0, 1 and 2 denote coating layer, substrate layer and thin-film layer respectively. By subtracting equation (4) from equation (3) the equation (5) is obtained. Then thermal conductivity of the thin film, λ_2 is given by equation (6).

$$(D_{012} - D_{01})(C_0 d_0 + C_1 d_1) + D_{012}C_2 d_2 = D_2 C_2 d_2$$
 (5)

$$\lambda_2 = D_2 C_2 = D_{01} C_1 \left\{ \frac{C_2}{C_1} + \left(\frac{C_2}{C_1} + \frac{C_0 d_0}{C_1 d_1} + \frac{d_1}{d_2} \right) \left(\frac{D_{012}}{D_{01}} - 1 \right) \right\}$$
 (6)

In general, the specific heat capacities per unit volume of solids do not differ significantly from each other and equation (7) is valid. Assuming that the thicknesses of the layers are related as shown in equation (8), (true for most practical cases), equation (6) can be simplified as equation (9). The error of λ_2 due to the approximation is estimated to be less than ± 1 %. It should also be noted here that the coating layer does not matter.

$$0.7 < \frac{C_2}{C_1}, \frac{C_0}{C_1} < 1.3 \tag{7}$$

$$\frac{d_0}{d_1} < \frac{1}{100}, \quad \frac{d_1}{d_2} > 100$$
 (8)

$$\lambda_2 = D_{01}C_1 \left\{ 1 + \frac{d_1}{d_2} \left(\frac{D_{012}}{D_{01}} - 1 \right) \right\}$$
 (9)

The thermal diffusivities of the deposited region (thin film + substrate), D_{012} and the non-deposited region (substrate), D_{01} can be determined by the measurement of a free-standing specimen. The thicknesses of the substrate and the thin film, d_1 , d_2 can also be determined by other means. If specific heat capacity per unit volume, C_1 of the substrate and the ratio of the specific heat capacity per unit volume, C_2/C_1 is known, then the thermal conductivity of the thin film, λ_2 can be determined. It should be noted here the required accuracy for the determination of the thicknesses of the substrate and the thin

film, d_1 , d_2 and the specific heat capacity per unit volume, C_1 is the same as that required for the determination of the thermal conductivity of the thin film.

The specific heat capacity per unit mass of a solid is the same regardless of its crystalline or other state, but the densities can differ depending on the amorphous or crystalline state and other factors. Assuming that there is no difference of chemical composition between the bulk material and the thin film and there are no significant vacancies in the thin film, the specific heat capacity per unit mass of the thin film will be same as that of the bulk material. Providing the density of the thin film can be determined by some technique, then its specific heat capacity per unit volume can be determined as the product of the measured density and the specific heat capacity per unit mass of the bulk material.

As mentioned earlier, specific heat capacities per unit volume of solids do not differ significantly and C_2 / C_1 is equal to 1 with an uncertainty less than ± 30 %. When thermal conductivity, λ_2 of a thin film is much greater than that of the substrate, it can be determined with its specific heat capacity per unit volume unknown.

On the other hand, the sensitivity of the measurement of thermal conductivity, λ_s depends on the thermal conductivity of the substrate, thickness ratio, d_1/d_2 and precision of the thermal diffusivity measurement of a free-standing specimen. At present the precision of the thermal diffusivity measurement is ± 0.3 %. Thus the sensitivity of the measurement of λ_s can be expressed by equation (10).

$$\lambda_s = D_{01}C_1\{0.3 + \frac{d_1}{d_2} 0.003\} \tag{10}$$

When the thickness ratio, d_1/d_2 is 100, the sensitivity of the thermal conductivity of a thin film is comparable with the expected maximum uncertainty of specific heat capacity per unit volume of the thin film, which is ± 30 %. When a borosilicate glass substrate is chosen, which has a specific heat capacity per unit volume of 1.80×10^7 J·m⁻³·K⁻¹ and a thermal diffusivity of 5.6×10^{-7} m²·s⁻¹, it can be calculated as ± 0.3 W·m⁻¹·K⁻¹. The sensitivity can also be expressed as the thermal conductance (W·K⁻¹) of the thin film, when the substrate material and its thickness are constant. When a borosilicate glass substrate with a thickness of 30 μ m is set, the sensitivity of the thermal conductance of a thin film is ± 90 nW·K⁻¹.

3. EXPERIMENTAL SYSTEM

The basic system and specifications have been described in detail previously and only salient features included here. [3, 12] The blackened surface (by using 1000 Å thick bismuth or 1 micron thick dry graphite film) of a test specimen of the order of $12.5 \sim 30$ mm long and $2.5 \sim 5$ mm wide by a thickness of $1 \sim 700$ µm with a thermocouple (0.1 mm diameter) fixed on the opposite surface is irradiated by thermal energy from a laser diode source.

As shown schematically in **Fig. 3** the modulated laser beam is focused to a spot on a 24 face polygonal mirror rotating at 6000 rpm and is concentrated into a $6 \sim 9$ mm long, 0.5 mm wide strip by using a scan lens. The resultant temperature signal is amplified using an input transformer and a low noise amplifier. The system is operated by a microcomputer connected to a personal computer with RS232C interface. A firmware program performs a Discrete Fourier Transformation and makes averaging the signal in the frequency range 0.01 to 10 Hz to provide the amplitude and phase data together with the spatial distance and temperature data to the personal computer. Analysis of the

resultant data delivered 1 data set per 10 seconds is carried out using the firmware. The software, which run on Windows95/98/NT/2000, gives separate value of D_a^* , D_p^* and D. For a measurement the thermal diffusion length is selected in the range of $1 \sim 3$ mm according to the length of the specimen and frequency, f set to this length by trial measurements. Actual measurements are made at frequencies of f/2, f and 2f with data collected and averaged for a large number of data points (more than 60) operating at a slow ramp rate. However, in the case when the frequency is lower than 0.05 Hz (for materials having very low thermal diffusivities and/or large thicknesses), measurements are undertaken at two discrete positions over long time intervals. In both cases the total measuring time is of the order of $10 \sim 20$ minutes. For the highest precision it is preferable to use vacuum conditions and these are necessary where materials have thermal diffusivity values below approximately 5×10^{-6} m²·s⁻¹. [13]

3. SPECIMEN PREPARATION

For the sub-micron thick film of AlN and Al_2O_3 a specially designed specimenholder-frame, shown in **Fig. 4**, is used to keep the very thin glass substrate from damage. The substrate and frame materials are borosilicate glass, D263 having a thickness of 30 μ m and 200 μ m respectively, supplied by DESAG (Schott Group). The substrate was machined to a width of 2.5 mm with a non-uniformity less than ± 0.3 % and 12.5 mm long. It is attached to the frame by polyimide adhesive and the whole specimen-holder-frame heat-treated up to 340 °C in air. The aluminum nitride films and aluminum oxide films was then deposited on a half surface of the substrate. The conditions of the composite specimen are listed in **Table I**.

The specimen-holder-frame was coated uniformly over the whole surface with 1000 Å thick bismuth by evaporation. Every measurement was carried out at room

temperature in a vacuum of less than 0.01 Pa using turbo molecular pump, to eliminate the effect of air layers. Frequencies of ac-calorimetric measurements were selected in the range of 0.1 Hz, so that the diffusion length is much longer than twice of the thickness of the specimen and shorter than 0.3 times of the half-length of the specimen. [14, 15]

4. PROPERTIES OF THE FILM

Deposition of the AlN and Al₂O₃ films were accomplished by means of magnetron sputtering with an inductively coupled r-f plasma with a sputtering target of aluminum (99.999 %) and gas partial-pressure ratio, N₂/Ar of 1/33. The properties of the aluminum nitride (AlN) films have been investigated by T. Morita, et. al. using the film deposited on silicon substrate by means of ellipsometry, RBS (Rutherford backward scattering), AES (Auger electron spectroscopy), XRD (x-ray diffraction), HRTEM (High resolution transmission electron microscope), (I-V) electrical resistance, etc. [14]

The results of the ellipsometry indicated that the refractive index, n was 2.05. The chemical compositions of the films determined by RBS are listed in **Table II**. In addition the element ratio, N/Al by AES was shown to be almost constant independent of the depth of the film. The (002) lattice plane spacing of AlN crystal was determined to 0.2498 nm, which is very close to the ASTM value of 0.2490 nm by XRD.

5. RESULTS AND DISCUSSIONS

The experimental data are plotted in **Fig. 5** illustrating the expected linear relationship for logarithmic amplitude decay and phase shift as a function of the spatial distance respectively. The homogeneity of the substrate was confirmed to be within ± 0.3 % by the measurement of specimen-holder-frames on both regions. Results of the apparent thermal diffusivities of the deposited region (thin film + substrate), D_{012} and

the non-deposited region (substrate), D_{01} at room temperature are listed in **Table III** Derived thermal conductivity of the aluminum nitride (AlN) film and aluminum oxide film (Al₂O₃) are listed in **Table IV**. The results indicate clearly that the thermal conductivity of materials in the form of very thin (sub-micron) films is significantly lower than that of the bulk materials. However very little difference between the values of AlN films and Al₂O₃ films was shown, not withstanding in the case of bulk materials. For high purity AlN and Al₂O₃ the bulk values are of the order of 180 ~ 250 W·m⁻¹·K⁻¹ and 30 W·m⁻¹·K⁻¹ respectively. These facts indicate that the thin films are in an amorphous rather than crystalline state. It was also shown that there is thickness effect indicating that the reduction of value could possibly be related to the crystal size affecting contact resistance between particles and/or due to difference of mean free path such that the scattering behavior is affected.

A chromatic thin film with thicknesses of 1000 and 2400 Å deposited on a half surface of PET (Polyethylene Terephthalate) substrate are also measured. This substance is used as a recording layer of CD-R media of the computer system. The conditions of the composite specimen are listed in **Table V.** Every measurement was also carried out at room temperature in a vacuum less than 0.01 Pa. Derived thermal conductivities are listed in **Table VI.** In this case no significant thickness dependence is found. This fact indicates that the films are primarily in an amorphous state.

On the other hand the results of the thermal conductivities of the PET substrate itself is $0.54 \sim 0.59 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, which is much higher than the value for bulk (solid) materials. Thermal conductivity of the polyimide film (50• m thick Kapton-H, supplied by Dupont-Toray Co.) are also measured on 6 different pieces or regions of the specimen. The polyimide film is also found to have higher thermal conductivity (0.74 \pm 0.02 W·m⁻¹

¹·K⁻¹) than the literature values obtained on bulk (solid) materials. In these experiments, the effect of the bismuth-layer was examined by making measurements at different thicknesses of the bismuth-layer and was confirmed that it is small enough. Assuming that thermal conductivity of the bismuth is as same as that of the bulk material (8.5 W·m⁻¹·K⁻¹), it is possibly much smaller; the effect is calculated to be less than 0.017 W·m⁻¹·K⁻¹. The effect of the air-layers was also examined by making measurements at different pressure points and was confirmed to be less than 1 %. In general, plastic films seem to have anisotropy in its thermal conductivity due to the process of making films.

Results of the derived thermal conductivity of a dielectric substance, ZnSSiO₂ with a thickness of 2.0• m, deposited on a half surface of the borosilicate glass (200µm thick) is quite low (less than 0.3 W·m⁻¹·K⁻¹). This substance is used as protective layers which sandwich the recording layer in CD-RW media of the computer system.

SUMMARY

A modified ac-calorimetry technique has been used successfully to measure the thermal properties of sub-micron films deposited on a homogenous by means of the differential method. The method is capable of measuring the thermal diffusivity of a free-standing specimen to a precision of ± 0.3 %. Results for AlN film and Al₂O₃ films having thicknesses from 1000 to 3000 Å deposited on glass indicate that the thermal conductivity is significantly lower than that of the bulk material. However there is very little difference between the values of the AlN films and Al₂O₃ films, notwithstanding in the difference in values of the bulk materials. The low values indicate that thin films are primarily in an amorphous rather than crystalline state. It was also shown that there is thickness effect indicating that the reduction of value could possibly be related to the crystal size affecting contact resistance between particles and/or due to difference in the

mean free path such that the scattering behavior is affected. Results for a chromatic substance having thicknesses of 1000 and 2400 Å deposited on PET substrate showed a low thermal conductivity, but no thickness dependence. This fact indicates that such films are also in an amorphous state.

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(Figure captions)

- Fig. 1 Schematic view for the setup of a free-standing specimen
- Fig. 2 Schematic view of the setup of the differential method
- Fig. 3 Optical arrangement of the laser-heating Ångstrom method
- **Fig. 4** The specimen-holder-frame for a thin film deposited on the half surface of a rectangular substrate.
- **Fig. 5** Experimental data illustrating the expected linear relationship for logarithmic amplitude decay and phase shift as a function of the spatial distance.
- **Table I.** Conditions of the composite specimen of AlN and Al₂O₃ thin films
- **Table II.** Chemical composition of the aluminum nitride (AlN) films determined by RBS.
- **Table III.** Results of the apparent thermal diffusivities of the deposited region (thin film + substrate), D_{012} and the non-deposited region (substrate), D_{01} at room temperature.
- **Table IV.** Derived thermal conductivity of aluminum nitride (AlN) and aluminum oxide (Al $_2$ O $_3$) thin film deposited on a borosilicate glass substrate.
- **Table V.** Conditions of the composite specimen of chromatic thin films.
- **Table VI.** Derived thermal conductivity of chromatic thin films deposited on PET substrate

Fig. 1

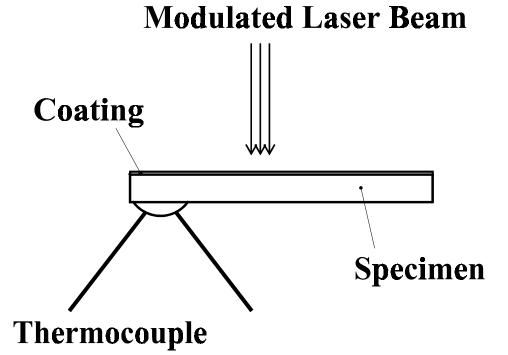


Fig. 2

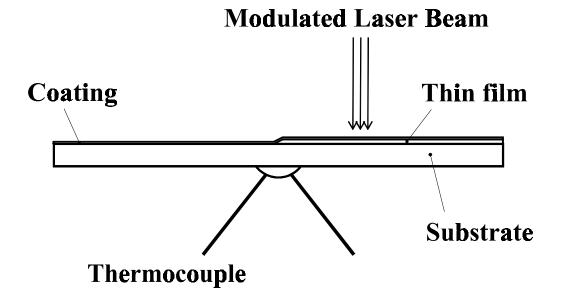


Fig. 3

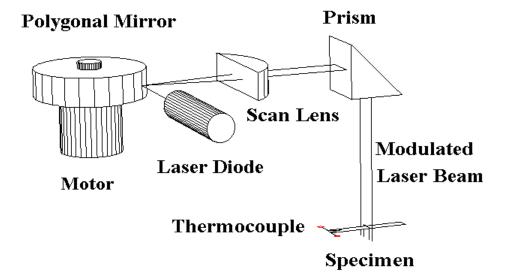


Fig. 4

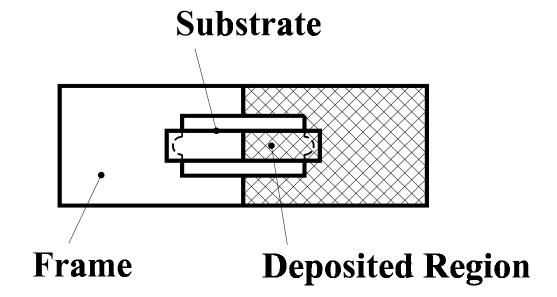


Fig. 5

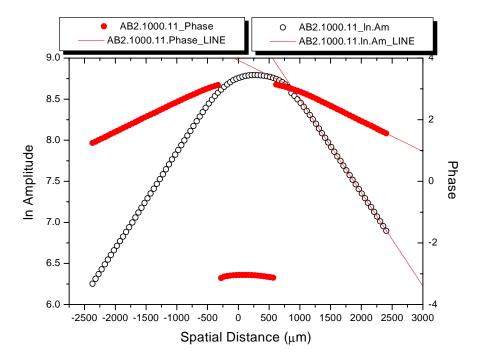


Table I

Sample name	Size (mm)	Thickness (µm)	Specific heat capacity (J·g ⁻¹ ·K ⁻¹)	Density $(\times 10^{-6} \text{ g} \cdot \text{m}^{-3})$	Supplier
Borosilicate glass (D 263)	2.5 × 10	30 ± 5	0.72 [16]	2.51	DESAG (Schott Group)
Aluminum nitride	2.5 × 10	0.1, 0.2, 0.3	*0.71 [16]	*3.26	T.Morita et. al. of
Aluminum oxide	2.5 × 10	0.1, 0.2, 0.3	*0.79 [16]	*3.99	Ulvac Japan Ltd.

^{*(}note) These assume that the materials are not highly porous or impure.

Table II

Substrate temperature	Element ratio	Yield Al: at 200 °C / Yield Al: at
(°C)	N / Al	RT
RT	0.969	1.04 ~ 1.05
200 °C	0.883	

Table III

Thickness (Å)	$D_{012} (\times 10^{-7} \mathrm{m}^2 \cdot \mathrm{s}^{-1})$	$D_{01} (\times 10^{-7} \mathrm{m}^2 \cdot \mathrm{s}^{-1})$	Literature Value of
			the substrate
			$(\times 10^{-7} \text{ m}^2 \cdot \text{s}^{-1})$
			by TPRC [17]
1000	5.62 ± 0.017	5.54 ± 0.004	5.5 (reported error
2000	5.85 ± 0.010	5.64 ± 0.010	~15 %)
3000	6.08 ± 0.015	5.67 ± 0.030	

Table IV

Sample name	Thickness (Å)	Thermal conductivity • W·m ⁻¹ ·K ⁻¹ •	Uncertainty (W·m ⁻¹ ·K ⁻¹)
Aluminum	1000	5.6	± 0.8
nitride	2000	6.6	± 0.6
	3000	8.4	± 0.35
Aluminum	1000	1.0	± 1.2
oxide	2000	3.3	± 0.6
	3000	4.5	± 0.45

Table V

Sample name	Size (mm)	Thickness (μm)	Specific heat capacity (J·g ⁻¹ ·K ⁻¹)	Density $(\times 10^{-6} \text{ g} \cdot \text{m}^{-3})$
PET	30 × 5	112 ± 2	1.18	1.6
No.1	15 × 5	0.110	Unknown	Unknown
No.2	15 × 5	0.240	Unknown	Unknown

Table VI

Sample name	Thermal conductivity • W·m ⁻¹ ·K ⁻¹ •	Uncertainty (W·m ⁻¹ ·K ⁻¹)
No.1	8.9	± 2.8
No.2	6.9	± 0.4